



# EUPT-FV21 SPECIFIC PROTOCOL

## European Union Proficiency Test for Pesticide Residues in Fruits and Vegetables (2019)

### Introduction

This protocol is complementary to the General Protocol of EU Proficiency Tests (EUPT) for Pesticide Residues in Food and Feed (8<sup>th</sup> Edition). This Proficiency Test is organised by the EURL for Pesticide Residues in Fruits and Vegetables covering Multiresidue Methods (MRM) of analysis.

According to Article 28 of Regulation 396/2005/EC (23<sup>rd</sup> February, 2005) of the European Parliament and of the Council, all laboratories analysing samples for the official control of pesticide residues shall participate in the European Union Proficiency Tests (EUPTs) for pesticide residues organised by the European Union.

These proficiency tests are carried out in order to improve the quality, accuracy and comparability of the residue data and to evaluate the laboratory capacity to report results that covers the entire range of maximum residue limits (0.005 - 15 mg/kg) in all groups of fruit and vegetable matrices (high water, acid and fat content). Bearing that in mind, a wide concentration range should be covered with the different analytes present in the test item.

### Test Item

This proficiency test is based on the analysis of pesticide residues in **red cabbage**. The cabbages were grown in a greenhouse in Almería. The pesticide treatments carried out were pre-harvest using commercial formulations and post-harvest using analytical standards. The test item was frozen (using liquid nitrogen), chopped, homogenised and sub-sampled into polyethylene bottles that had previously been coded.

Ten of these bottles containing the test item were chosen randomly and analysed to check for homogeneity.

The test item is stored frozen (-20°C) prior to shipment to participants.

Six bottles, again chosen randomly, will be analysed over a period of time to confirm the stability of the pesticides in the test item (three when the test items are shipped,



then other three bottles a few days after the deadline for submitting results). There will be one further analysis during this period using three bottles more and reproducing the sample shipment to see if there is any degradation of any of the pesticides present in the test item.

Together with the test item, a blank sample of red cabbage homogenate will be sent to the participants. The blank material should not contain any of the pesticides in the Target Pesticides List, at or above, the specified MRRLs. If a pesticide is identified by the Organisers in the blank material and there is no possibility to find a different blank, that pesticide could be removed from the Target Pesticides List and it will be communicated to all the participants by e-mail.

### **Amount of Test Item**

Participants will receive:

- Approximately 200 g of red cabbage test item treated with pesticides.
- Approximately 200 g of 'blank' red cabbage test item.

### **Shipment of Test Item**

All Test Items will be frozen and packed in polystyrene boxes surrounded in dry ice and packed into cardboard boxes.

The shipment of the test items will be carried out over a one-week period from the 25<sup>th</sup> February 2019. The Organiser will try to ensure that all the packages arrive on the same day to each laboratory. An information message will be sent out by e-mail before shipment. Laboratories must make their own arrangements for the receipt of the package. They must inform the Organiser of any public holidays in their country/city during the delivery period given in the calendar, as well as making the necessary arrangements for receiving the shipment, even if the laboratory is closed.

The Organisers will not take the responsibility for a parcel if it is retained at customs.

### **Advice on Test Item Handling**

Once received, the test item should be stored deeply frozen (-18°C or less) prior to analysis thus avoiding any possible deterioration/spoilage. The test item should be mixed thoroughly before taking the analytical portion(s).

All participants should use their own routine standard operating procedures for extraction, clean-up and analytical measurement and their own reference standards for identification and quantification.



### **Subcontracting**

All analytical determinations concerning the test item treatment analysis will be performed in a laboratory which is ISO 17025 accredited, and which has been previously evaluated by the Organisers.

### **Target List**

In addition to the pesticide target list of mandatory compounds, a "voluntary target list" containing pesticides which might be present in the test item will be published. Those voluntary pesticides will not be used for the evaluation of the laboratories into Category A or B, and a separate statistical evaluation will be made for them.

### **Assigned value and robust relative standard deviation**

In order to minimise the influence of out-lying results on the statistical evaluation, the assigned value will be estimated using the robust statistics as described in ANNEX C of ISO 13258:2015, where the robust mean ( $x^*$ ) according algorithm A is defined. For the calculation of the assigned value only results reported by EU and EFTA countries laboratories will be taken into account.

Also, the robust relative standard deviation ( $CVs^*$ ) will be calculated for each analyte.

### **Laboratory assessment**

For the assessment of the overall laboratory performance, the Average of the Squared z-Score ( $AZ^2$ ) will be used as in the last Proficiency Test, but only for those laboratories in Category A, which will be those laboratories that are able to analyse at least 90% of the pesticides in the target list, that are able to detect at least 90% of the pesticides present in the test material and that report no false positives. Within Category A, the laboratories will be sub-classified as "good", "satisfactory" or "unsatisfactory". All the other laboratories will be classified in Category B. This information will be available in the General Protocol.

### **Steps to follow**

This Proficiency Test will be made up of the following nine essential steps:

1. To participate, each laboratory must complete the Application Form on-line, whose link is available on the EURL-FV Web page, before the deadline stipulated on the Calendar. It is recommended that laboratories download the Target Pesticide List from this web site. Laboratories should carefully read the Target Pesticide List, where the



Minimum Required Reporting Limits (MRRLs) are given. The MRRLs do not always correspond with the EU MRLs set for red cabbage.

2. When the registration period is closed, laboratories will receive an e-mail with their user name and password for the restricted area of submission of results.

3. The sample delivery will be **250 euros** for EU/EFTA participants and **350 euros** for participants from other countries. The laboratories will receive an invoice and after that they can start the payment procedure. An e-mail showing the bank transfer confirmation, or similar, may be requested at any time by the Organiser. **Payments without a Laboratory Code identifying them will not be considered as paid.**

4. Any request, clarification, complaint, suggestion or comment should be made using a **Contact Form** placed in the restricted area of the Proficiency Test webpage.

5. **Scope Form** will be placed in the restricted area and will be open to participants from the 11<sup>th</sup> February – 22<sup>nd</sup> February 2019, prior to test item shipment. The aim is that laboratories provide information regarding their scope of analysis before receipt of the test item. As default, all compounds of the target lists are selected and the MRRL is listed in the scope. Laboratories will be asked to deselect the compounds they will not include in their PT scope and insert their Reporting Limits for each pesticide. If a laboratory does not select their scope, the default values will be considered for its evaluation.

6. When the participant laboratories receive the test item (and not before), they must enter the restricted area again and submit the **Test Item Receipt Form** to inform the Organiser that they have accepted the test item. If no test item has been received by 1<sup>st</sup> March 2019, the laboratories should contact the Organiser using the Contact Form of the restricted area. If the test item receipt form is not filled in, the Organiser will consider that the participant has accepted the test item.

7. Once the laboratory has analysed the test item and is ready to submit their data, they must enter their results at various steps by accessing the restricted area in the EURL-FV web site. The participant laboratories must respect the deadline for submitting their results – 25<sup>th</sup> March 2019- using the tabs **Detected, Edit results and Edit Methods** on-line.



For each pesticide included in the laboratory scope, the Reporting Limit (RL) will be requested. The MRRL and the participant's own RL will be used to help identify and calculate z scores for false negative results. This form will also request information on which of the pesticides sought by the laboratory is within the laboratory's routine scope and whether it is accredited.

The laboratories will be also asked to report any pesticide that may have been detected in the blank test item.

All concentrations must be expressed in mg/kg together with the recovery as a percentage. The actual results/residue levels measured must be reported as numbers. Symbols (>, <, ±, ≥, ≤, ...) will not be accepted. **IMPORTANT:** If your result is not correctly expressed it will be considered as 'ND' (Not Detected).

The number of significant figures should be based on the procedures provided in SANTE/11813/2017. Additional significant figures may be recorded for the purpose of statistical analysis.

Results should not be reported where a pesticide was not detected or was detected below the laboratory LOQ. In both cases, this will be recorded as 'ND'. If a pesticide was not sought, it will be recorded as 'NA' (Not Analysed). **If a laboratory fills in the scope form, but it does not report results neither fills in the methods form, all the pesticides will be considered as NA.**

The laboratory will also be asked to report the details of the analytical methods they used. A list including all the pesticides detected in the sample will be shown along with a pesticide reference number. Laboratories may describe a method for the first pesticide and use this pesticide reference number to refer to other pesticides determined using the same method.

When all fields are filled out, laboratories must accept and submit their final results by clicking the check box and then click on Final submission, before 25<sup>th</sup> March 2019.

**IMPORTANT: After the final submission it will NOT be possible to edit the results.**

Participants will receive an email confirming the submission of their results, and with an attached excel file with their submitted data.

It should **not** be assumed that only pesticides registered for use on red cabbage are present in the test item.

8. One final tab, **Additional Info**, will be accessible after the deadline for submission of results has passed. In this Form it will be possible to submit the method information of false negative results. It will be available from 25<sup>th</sup> March – 1<sup>st</sup> April 2019. Not all laboratories may need to fill this in. It will depend upon information reported on previous Forms.

9. The Organiser will evaluate the results at the end of the proficiency test, once the deadline for receipt of results has passed. The Organiser will send all the participants the preliminary results, containing all the reported concentrations, in order to make sure that there was not any mistake from the Organisation side. When necessary, the Organiser will ask the participants by e-mail specific details about the methods of analysis used. After a stipulated deadline, the results will be evaluated and a preliminary report containing the preliminary assigned values and z scores will be sent to the participants. Finally, after evaluation by the Scientific Committee, the Final Report will be published online and a copy will be sent to each participant laboratory. This report will include information regarding the design of the test, the homogeneity and stability results, a statistical evaluation of the participant's results as well as graphical displays of the results and any conclusions. Results submitted by non-EU/EFTA laboratories might not always be used in the tables or figures in the final report. Further relevant information considered to be of value may also be included.

### Calendar

EUPT-FV21 CALENDAR	
Activity	Date
Registration period at <a href="http://www.eupt-registration.eu">www.eupt-registration.eu</a>	20 <sup>th</sup> December 2018 -25 <sup>th</sup> January 2019
Specific Protocol published on the Web site.	11 <sup>th</sup> February 2019 at the latest
Selection of the scope.	11 <sup>th</sup> - 22 <sup>nd</sup> February 2019
Sample distribution.	25 <sup>th</sup> February 2019
Deadline for receiving sample acceptance	4 <sup>th</sup> March 2019
Deadline for receiving results	25 <sup>th</sup> March 2019
Filling in additional information, if necessary.	25 <sup>th</sup> March - 1 <sup>st</sup> April 2019
Preliminary Results: only results, no statistical treatment.	April 2019
Preliminary Report: (containing preliminary assigned values and z scores)	May 2019
Final Report distributed to the Laboratories.	Final Report distributed to the Laboratories.

**Cost of test item shipment.**

**EU/EFTA** laboratories will be charged **250 €** for the shipment cost, for **non-EU/EFTA** laboratories the amount will be **350 €**. Regarding payment procedures - each laboratory can specify their details and invoice requests when applying for the test.

**Please, do not pay for this EUPT until we send you the invoice.**

**Remember to include your Laboratory Code in the subject of the bank transfer.**

Payment details are as follows:

**BANK NAME: CAJAMAR - Caja Rural Sociedad Corporativa de Crédito**  
**BANK ACCOUNT HOLDER: Universidad de Almeria**  
**BANK ADDRESS: Office Number 990. Universidad de Almeria. Spain**  
**ACCOUNT NUMBER: ES0730580130172731005000**  
**SWIFT: CCRIES2A**

**Contact information**

The official organising group details are as follows:

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**Quality Control Group**

Dr. Antonio Valverde, University of Almería, Spain  
 Dr. Paula Medina, European Food Safety Authority, Italy.

**Advisory Group**

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 Dr. Miguel Gamón, Laboratorio Agroalimentario, Valencia, Spain.  
 Dr. Magnus Jezussek, Bavarian Health and Food Safety Authority, Erlangen, Germany.  
 Dr. André de Kok, NVWA, Wageningen, The Netherlands.



Mr. Ralf Lippold, CVUA, Freiburg, Germany.

Dr. Sonja Masselter, AGES GmbH Institute for Food Safety, Innsbruck, Austria.

Mr. Finbarr O'Regan, Pesticide Control Laboratory, Celbridge, Ireland.

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Dr. Tuija Pihlström, National Food Agency, Uppsala, Sweden.

Dr. Mette Erecius Poulsen, DTU, Copenhagen, Denmark.